Status Report on the Rebuilding of Beam line 10.3.2 for Elemental Mapping and Micro X-Ray Absorption Spectroscopy.

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INTRODUCTION

In FY99 this beam line was used by a wide range of users (over 70) from many disciplines to carry out the techniques of elemental mapping and micro X-Ray Absorption Spectroscopy (XAS) on a wide range of samples.

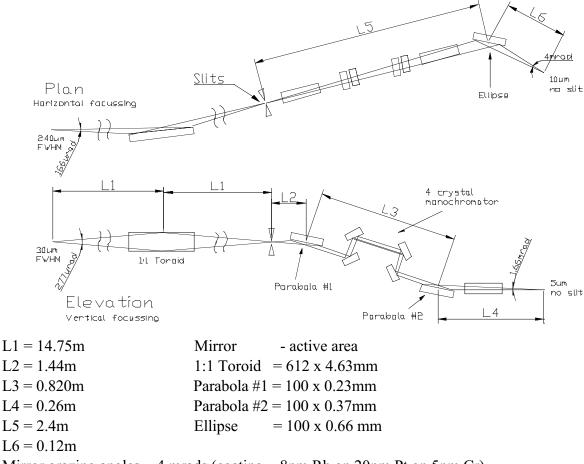
Experiments were carried out with samples in their natural state either in air or in a simple wet cell. Elemental distribution is carried out by rastering the sample in front of a monochromatic (5-14KeV) micron sized x-ray beam and the characteristic x-ray fluorescence detected with a solid-state energy resolved detector. Areas of interest are established and these areas can be probed with micro-XAS, which allows for speciation of the element of interest. The energy range was suitable for the transition metals, where they can be a minor species. In non-uniform environments where spatial inhomogeneity is of the same order of magnitude as that of the beamsize (<5 microns) such systems are ubiquitous in the three broad categories of biological, earth/environmental and material sciences. To date there have been few tools to investigate such systems and none of which could be regarded as ideal. Micro-XAS when combined with fluorescence detection has the capability of providing previously unattainable insights into the structure and chemistry of a species and how it is related to the function of that species and others that may coexist in such complex systems.

The equipment used was sub optimal development equipment whilst routes to funding were being organized. In FY2000 UCDRD and LDRD capital funds allowed a start to be made on upgrading this beamline to a fully user friendly, high flux, high throughput beamline. The aim is to build a world-class state of the art micro-XAS beamline available for this broad user community. Elemental scan times would drop from 10 hours with the demonstration system to 10 mins. Micro XAS scans would drop from 20 hours to 20 mins.

BEAMLINE LAYOUT

The schematic layout of the proposed upgraded beamline is shown in Figure 1. A more detailed description is given in ref [1]. The concept of the beamline is to have the 1:1 toroidal mirror relay the source to the adjustable slits, which then act as an adjustable sized source for the following optics. In this way the source is then effectively much closer to the sample with a consequent increase in flux. The spot size is dictated by the size of the adjustable slits, thus allowing spot size to be traded for flux. This will allow for rapid scanning over a large area of sample with a large beam (~10 microns) to find areas of interest followed by a fine scan with a smaller beam (down to ~0.25micron). The additional parabolic mirror after the slits is to allow illumination of the monochromator with parallel light. This will allow the full vertical acceptance (0.2mrad) of the beamline rather than with the test setup which has restricted the vertical

acceptance to the angular acceptance of the 4 crystal monochromator (~<0.05mrad). The increased flux requires improved detector and scanning stages. A new multi-element detector with a X5 increase in solid angle of collection with fast processing electronic package is required. A new backlash free XY scanning stage will also be required to allow for rapid scanning in a serpentine manner. The detector and stage need to be integrated with the existing developmental software to produce a robust user friendly package.



Mirror grazing angles = 4 mrads (coating = 8nm Rh on 20nm Pt on 5nm Cr)

Fig. 1 Proposed optical layout for an optimized μ-XAS system on beamline 10.3.2

The upgrade requires that 20 m of beamline between the shield wall and hutch be replaced. The M1 toroid mirror in the middle. The entire experimental setup within the hutch will also need to be replaced. The hutch and front end are left unaffected.

PROGRESS

All the hardware and instrumentation for this facility has been designed and built, or purchased and installed. This includes :-

- All the optical and engineering design work.
- Fabrication, installation and testing of the figure of the main toroidal mirror M1
- Fabrication, installation and testing of the slit assembly

- Fabrication, installation and preliminary testing of the end station which includes the monochromator, focusing mirrors and vacuum housing
- Commissioning of the new 7 element Germanium solid state detector
- Commissioning of the new fast XY scanning stages.

The M1 mirror has the ability to image the electron source with minimal imperfections which has allowed the source size to be accurately measured for the first time on a dot 3 bending magnet source. The source size was found to be 40 x 290 um (FWHM VxH) in reasonable agreement with what was predicted. However this mirror will allow for another measurement to be carried out during ALS ring optimization. Current work on the end station involves the integration of the detector software and the fast scanning stage software.

REFERENCES

1. Upgrading of the Micro-X Ray Absorption Spectroscopy Beamline 10.3.2 at the ALS. A.A.MacDowell, H.A.Padmore, R.Celestre, G.M.Lamble, A.C.Thompson, D.Cambie, LSBL-539, Jan 2000

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